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# INJECTION TESTS AND EFFECT ON MICROSTRUCTURE AND PROPERTIES OF ALUMINIUM 7075 DIRECT THERMAL METHOD FEEDSTOCK BILLETS

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**Abstracts:** The success of semi-solid metal forming is dependent on a globular solid grain formation within a liquid phase. This paper presents experimental works concerning semi-solid metal processing of aluminium 7075 feedstock billets which were produced by direct thermal method. The flowability of feedstock billets was evaluated by an injection test processing unit. The feedstock billets were heated to a temperature of 620 °C by using a box furnace before transferred into a forming die. The formed feedstock billet was removed from the forming die after it was cooled to ambient temperature. Several analyses were conducted on the formed feedstock billets including dimensional measurement and microstructure analysis. The results show that the feedstock billets which contained the highest amount of free secondary phase were most successfully formed. Microstructure analysis results also revealed the formation of more globular and larger  $\alpha$ -Al solid grains in the same feedstock billets. In this experimental work, the feedstock billets with higher amount of secondary (liquid) phase had a significant effect on formability. It is concluded that in order to achieve successful formability of the direct thermal method feedstock billets, the billets need to have higher secondary phase content. Thus, important preparation methods of feedstock billets were characterised in order to allow for SSM processing.

## Introduction

There are two possible methods which are used in semi-solid metal (SSM) processing consist of rheo and thixo routes. In rheo routes, raw material which is originally from ingot processes without an intermediate solidification step. The molten metal which is slightly above the liquidus temperature is poured into a steel crucible and then treated to form a globular microstructure before moving into a forming machine. Thixo routes on other hand cooled the raw material into semisolid state into a die cavity with an intermediate solidification step. Thixo routes required preparation of SSM feedstock which has been initially treated in such a way when it is heated to semisolid range temperature it will form globular microstructures [1, 2].

There are several methods which are used to form a non-dendritic feedstock for SSM processing. Mechanical Stirring (MS) used augers, impellers or multiple agitators which attached to a rotating shaft and stirred the molten melt in order to retard the formation of the molten alloy from a dendritic to a globular microstructure [3]. This method is a cost effective method to be used for laboratory feedstock production and investigation. Swirled Enthalpy Equilibrium Device (SEED) process uses a metallic container to create a rapid thermal equilibrium condition in order to form a large number of nuclei within the bulk metal. This process can be used to produce globular feedstock of over 18 kg of product weight [4]. Low

Superheat Pouring with a Shear Field (LSPSF) process manipulates the solidification condition to control nucleation formation, nuclei survival and grain growth. The melt is poured into a rotating stainless steel barrel which is temperature controlled via simultaneous induction heating and air cooling in order to achieve a rapid cooling and produce a non-dendritic feedstock [5]. Direct Thermal Method (DTM) allows the cooling of superheated alloy within a thin walled copper mould. This initial rapid cooling gives rise to copious nucleation which contributes to a globular feedstock [6].

An important metallurgical characteristic that has a significant effect during SSM processing is a fraction solid. Research shows that it is important to obtain a low viscosity and a high fraction solid in SSM processing [7, 8]. The fraction solid determines the flow ability of the material and influence the formation of the microstructure and defects. The low viscosity component helps material to flow inside die cavity and high fraction solid helps to prevent various defects, finer internal structure and high quality products. The suitable fraction solid volume within material helps the grains to rotate, slip and move freely under a small external working force. Lashkari and Ghomashchi [8] suggest the SSM processing of the feedstock to obtain a low viscosity and a high fraction solid content. The viscosity helps the material to flow inside the mould cavity meanwhile high fraction solid prevent the possible defect to deform.

Recent developments in SSM processing have heightened the need for able to process wrought aluminium alloys especially 7075 in SSM range which gives a significant affect to the performance of the formed part. There is a lack of detailed information within the literature for the thixoforming of 7075 which used feedstock billets from the DTM. This paper will focus on the design of injection test processing unit and processing of SSM feedstock billets within semisolid range temperature. This paper also attempts to show the evaluation of SSM feedstock billet towards its formability. The relationship between microstructure and formability of SSM feedstock billets was also analysed.

## Material and Procedures

The chemical composition of aluminium 7075 alloy which was used in this work, as determined with Optical Emission Spectroscopy, Foundry Master Oxford Instruments is presented in Table 1.

Table 1: The chemical composition of the aluminium 7075.

| Source (wt%) | Al   | Cr  | Cu   | Fe   | Mg   | Mn   | Si   | Ti   | Zn   |
|--------------|------|-----|------|------|------|------|------|------|------|
| Experiment   | 88.5 | 0.2 | 2.02 | 0.24 | 2.38 | 0.12 | 0.14 | 0.09 | 6.04 |

Design and construction of injection test processing unit consists of four main components. The components were hydraulic press machine, upper plate, lower plate and forming die. Schematic view for this machine is presented in Figure 1. Hydraulic systems were used to produce pressing force. The machine was designed in a way that the pressing force was applied from top to bottom. Maximum machine pressure capacity was 70 bars controlled by switch panel on top of the machine. The machine was also equipped with six K-type thermocouples which connected to a machine controller by using a 1 m wire. One of the thermocouple then was placed into the forming die through forming die inlet to measure die temperature during heating. Two controlled switches equipped with a sensor as a machine guarding to ensure the safety of operator. The stainless steel bar was also mounted at this machine as the base for the die. The function of the upper plate was to assemble several parts consist of a plunger and guide posts. The plunger was used as a pushing rod in order to form feedstock billets. The forming die consists of upper part which was used as the incoming area

for the feedstock billets. Another part of the forming die was a lower part which the feedstock billets exit and formed. High resistance temperature tool steel was used as the material for this injection test processing unit.

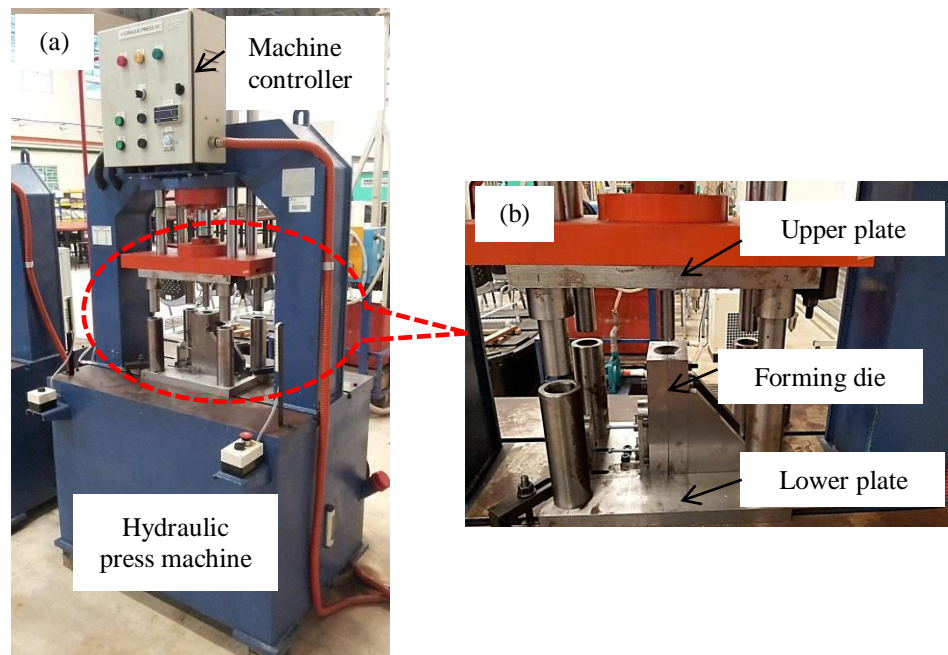


Figure 1: Schematic view of injection test processing unit which was used in this work with (a) overall view of hydraulic press machine and (b) detail views for upper plate, forming die and lower plate.

The aluminium 7075 feedstock billets were used in this experimental work initially processed by using DTM. Detail explanation about the method to produce feedstock billets were discussed in another publication [9]. Dimension of this feedstock billet was 70 mm in height and 22 mm in diameter.

The feedstock billet was placed into a 35 mm diameter, 50 mm height steel crucible. The crucible then was placed into a box furnace heated to a temperature of 640 °C and remained constant at that temperature for 30 minutes. Forming die at the injection test processing unit was heated to a temperature 115 °C by using a flammable torch. Following this, the steel crucible that contained feedstock billet was transferred out from the furnace to the forming die after 30 minutes. The feedstock billet was then poured out from the steel crucible into the forming die. Transport period for this operation was measured. The press switch that controls the upper plate into the lower plate movement was pushed to start forming operation. Once feedstock billet and forming die were at room temperature, the feedstock billet was removed from the forming die. Finally, the dimensions of the formed feedstock billet were measured by using a height gauge.

Metallographic samples were sectioned by using an alumina Buehler cut off wheel 245 mm in diameter and 1.78 mm thick with coolant. Samples from the formed feedstock billets were cut at conical area located 5 mm from the end of feedstock billets. The samples were mounted in Bakelite resin by using Buehler Simplimet 2000 Mounting Press hot mounting machine. Mounted samples were ground by using 240, 600, and 1200 grit size silicon carbide (SiC) at 0.1 MPa (1 bar) pressure and at a 200 rpm grinding wheel speed. Ground samples were then polished by using Textmet 1000 cloths with 9 micron, 6 micron, 3 micron size diamond paste and given a final polish to 0.05 micron size alumina polishing suspension for 4 minutes each, at 0.1 MPa (1 bar) pressure and the polishing speed of 150 rpm. Buehler

Motopol 2000 grinder/polishing machine were used to perform both of grinding and polishing work. The samples were then etched using Keller's etch for approximately 20 seconds. Reichert ME F2 universal camera optical microscope was used to view the microstructures. Buhler Omnimet Enterprise software was then used to capture the microstructure images.

Grain size area, perimeter, circularity, diameter, and aspect ratio measurements were determined by using Image J software. The circularity is an indication of a perfect circle which occurs within a microstructure. The value that approaches at 1.0 is considered as the perfect circle with decreasing number toward 0.0 is indicated an increasing elongated shape. The aspect ratio is an indication for circular or square morphology which aspect ratio value increases with an elongated particle. Circularity of solid phase particle was calculated as four times pi divided by the particle perimeter squared ( $4\pi/P^2$ ) and the as aspect ratio was calculated as the major axis divided by the minor axis.

## Results and Discussion

### *Injection Test*

Figure 2 illustrates several examples of the feedstock billets which were formed after the injection test. This figure gives a better understanding about the measurement of overall length A and B. As shown in Figure 2, there is a significant difference between the formed feedstock billets at conical section that indicate the successful of injection test.



Figure 2: Several example of the formed billets which occurred after the injection test. The overall length of the billets was measured from bottom to maximum height of the feedstock billets.

The average injection test results for 10 feedstock billets which were formed after injection test are shown in Table 2. The results consist of overall length A and B after and transfer time during the injection test. The feedstock billets were processed initially with 10 different processing parameters which repeated three times in order to gain repeatability of the results. It is apparent from Table 2 that the average overall length A of the feedstock billets was changed after injection test. The feedstock billets decreased approximately 29 % in overall length compared with the original length. Meanwhile, average diameter for the feedstock billets were increased by 18 % compared with original billet diameter.

In order to get clear information about the injection test, the results were summarised and presented in Figure 3. The results show that the billets which were grouped in sample number 3 represent the highest value of overall length A-B. This shows that billets in sample number 3 were effectively filled into the conical area of die which indicates the ability of the feedstock billets to form. The feedstock billets in sample number 3 were the highest in formability compared with other feedstock billets. The feedstock billets in sample number 2

show the lowest overall length A-B which indicates the lowest formability during the injection test.

Table 2: The average overall length A, overall length B and transfer time results for injection test for 10 samples.

| Sample No. | Feedstock Billets Processing Conditions |                  | Average Overall Length A (mm) | Average Overall Length B (mm) | Average Transfer Time (s) |
|------------|---|------------------|-------------------------------|-------------------------------|---------------------------|
|            | Pouring Temperature (°C)                | Holding Time (s) |                               |                               |                           |
| 1          | 685                                     | 60               | 53.12                         | 47.04                         | 10.52                     |
| 2          |   | 40               | 51.71                         | 46.73                         | 11.87                     |
| 3          |   | 20               | 54.77                         | 44.19                         | 12.05                     |
| 4          | 665                                     | 60               | 51.27                         | 43.86                         | 11.03                     |
| 5          |   | 40               | 50.77                         | 45.55                         | 12.76                     |
| 6          |   | 20               | 51.96                         | 47.07                         | 11.38                     |
| 7          | 645                                     | 60               | 51.87                         | 46.41                         | 10.71                     |
| 8          |   | 40               | 51.75                         | 46.43                         | 10.30                     |
| 9          |   | 20               | 52.69                         | 47.45                         | 11.96                     |
| 10         | 685                                     | -                | 53.18                         | 47.45                         | 10.63                     |

Transfer time represents the duration of feedstock billets after transported out from furnace until the start button at the machine was triggered. This transfer time was used to determine the actual temperature of feedstock billets at the initial stage of injection test. Base on separate experimental work, it shows that cooling rate during the time of feedstock billets transferred out from the furnace was at 0.88 °C/s. The temperature at the respective time (base on transfer time) was calculated by using this cooling rate. The feedstock billets temperature after transported out from furnace was at 620 °C. The temperature for sample number 3 during the start button was triggered which calculated based on 0.88 °C/s cooling rate was approximately at 609 °C. The temperature then was used to estimate fraction solid within feedstock billets.

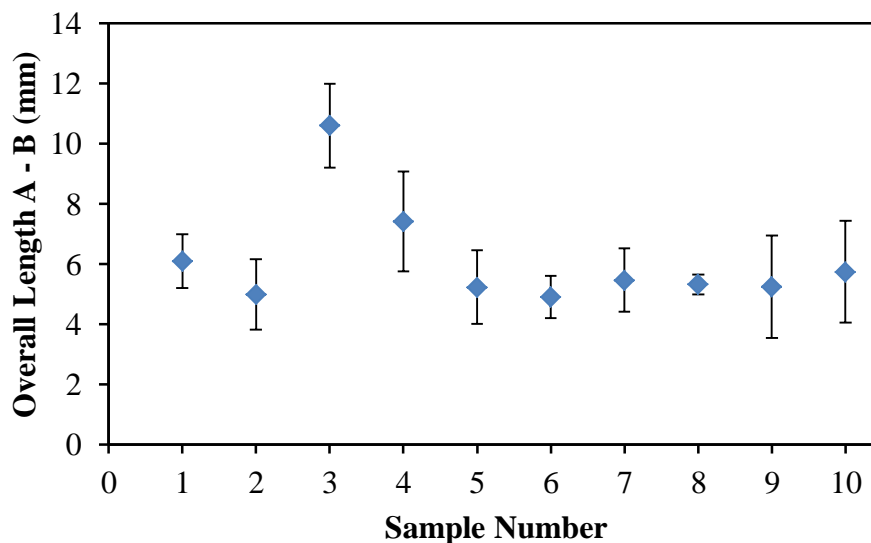


Figure 3: Overall length results for 10 group samples which measured after injection test (errors are 95% confidence intervals).



### Microstructure

Changes in microstructure of aluminium 7075 between initial feedstock billet after DTM and injection test are compared in Figure 4. The general microstructure formation after injection test were more globular and larger in size compared to initial feedstock billet (after DTM). The microstructure in Figure 4 (b) consists of Al solid grain which is surrounded with the secondary (liquid) phase. These results show microstructure one of the sample for sample number 3 which exceed the highest average overall length A that become more globular with higher average grain size after the feedstock billets were re-heated to semisolid range temperature.

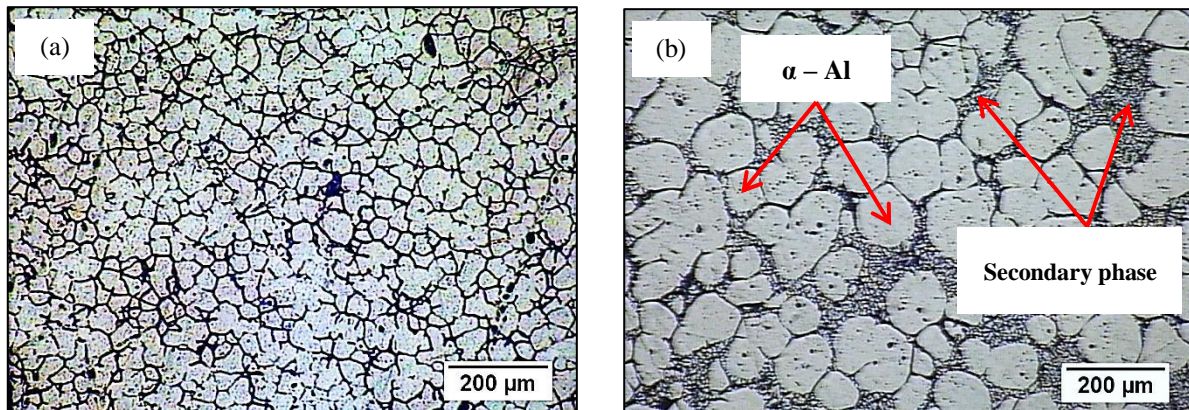


Figure 4: Microstructure aluminium 7075 for the feedstock billets sample number 3 with (a) initial feedstock billets after DTM and (b) after injection test.

Table 3 shows average grain size measurements for microstructure of sample number 3 consists of area, perimeter, circularity, diameter and aspect ratio. It is apparent from this table that microstructure formations after injection test were more globular and larger in size than microstructure before injection test. The average grain circularity was increased to 23 % and average grain diameter was increased to 58.7 % compared to initial feedstock billet before the injection test.

Table 3: Average grain size measurements for microstructure of sample number 3 before and after injection test.

|                              | Area<br>( $\mu\text{m}^2$ ) | Perimeter<br>( $\mu\text{m}$ ) | Circularity | Diameter<br>( $\mu\text{m}$ ) | Aspect<br>Ratio |
|------------------------------|-----------------------------|--------------------------------|-------------|-------------------------------|-----------------|
| <b>Before Injection Test</b> | 3156.21                     | 282.54                         | 0.52        | 86.66                         | 1.52            |
| <b>After Injection Test</b>  | 9916.42                     | 434.40                         | 0.64        | 137.50                        | 1.36            |

### Effect of properties of feedstock billets on Injection Test

A strong relationship was found between different types of feedstock billet used and average overall length A on injection test results. The injection test results showed that feedstock billets in sample number 3 were better in results compared with other feedstock billets. This result may be explained by the fact that feedstock billet microstructures in sample number 3 consist of higher secondary phase which occurs around solid aluminium particles. During the initial stage of feedstock billets preparation, feedstock billets in sample number 3 were quenched from the liquidus condition (approximately at 650 to 660 °C). This rapid cooling significantly affected the microstructures of the feedstock billet during reheating process. Grain boundaries were penetrated by the liquid matrix during reheating process due to dissolution of eutectic phase. At this time, grain spheroidization and



coarsening were started when liquid phase at grains boundaries. The higher secondary phase which was initially existed inside feedstock billets in sample number 3 catalysed this formation around solid aluminium particles. The higher secondary phase content inside feedstock billets in sample number 3 during processing assisted the microstructures to slip between others and easily formed to die cavity. This produced a significant effect to the injection test. This was due to solid fraction within material that was found to be one of the foremost factors in determining the success of SSM processing [10].

#### *Effect of the Injection Test on Microstructure*

It is apparent from Figure 4 and Table 3 that the microstructures of aluminium 7075 feedstock billets were changed after the injection test. Possible explanations for this might be the heating process of feedstock billets before and during the injection test. The feedstock billets were heated to semisolid temperature range and during this time eutectic phase which is the lowest melting temperature melted first. This reaction was believed to be occurred at 477 °C [11]. The liquid phase infiltrated solid particles which split microstructure into several large grains which known as primary  $\alpha$ -Al as in Figure 4. During this heating process it has been seen that primary  $\alpha$ -Al was surrounded by the eutectic liquid phase. As the temperature increased, fraction liquid increased and fraction solid decreased in feedstock billets microstructure. This resulted the melting of sharp corner of grains and produced more globular structure of primary  $\alpha$ -Al in the secondary (liquid) phase. This is evident in Figure 4 (b) which contains sharp corner of the primary  $\alpha$ -Al seems separated to smaller grains. The findings of the current study are consistent with most of the results reported in literature which found final microstructure of SSM slurry by thixoforming consists of primary  $\alpha$ -Al grains surrounded with secondary (liquid) phase [12, 13].

During the time feedstock billet was transferred into forming die, the feedstock billet temperature was approximately at 609 °C which is related to a 0.6 fraction solid that is an evidence of presence of high secondary phase content [11]. Rapid nucleation occurred between the cold surface of die and liquid phase of the feedstock billets results a smaller grain structure as in Figure 4 (b). As the initial microstructure of feedstock billets in sample number 3 were surrounded by higher secondary phase content, it catalysed the formation of smaller  $\alpha$ -Al solid grains. Detail explanations about this phenomenon were discussed in several publications [14, 15].

#### **Conclusion**

This paper has presented the determined important characteristics of DTM aluminium 7075 feedstock billets which were processed by using an injection test unit within the semisolid temperature range. The injection test which determines the formability of material gives a significant indicator for the successful test. The experimental results show that formability of the feedstock billets was influenced by secondary (liquid) phase content within the starting material. The feedstock billet of sample number 3 showed the best results from the injection test. The microstructure, as examined after injection, showed the formation of primary and smaller  $\alpha$ -Al solid grain surrounded by secondary (liquid) phase within sample that assists the feedstock billet formability. The transfer time affects the fraction solid within the material. The transfer time needs to be kept as low as possible to avoid excessive heat lost by convection. Based on experimental results, it can be concluded that in order to get the successful formability of DTM feedstock billets, the billets need to comprise higher secondary (liquid) phase content. This is a critical parameter in order to achieve an effective formability in SSM processing.

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